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Abstract. Solar dynamic (SD) space power systems require durable, high emittance surfaces on a number of critical components, such as heat receiver interior surfaces and parasitic load radiator (PLR) elements. To enhance surface characteristics, an alumina-titania coating has been applied to 500 heat receiver thermal energy containment canisters and the PLR of NASA Lewis Research Center's (LeRC) 2 kW SD ground test demonstrator (GTD). The alumina-titania coating was chosen because it had been found to maintain its high emittance under vacuum ($\leq 10^{-6}$ torr) at high temperatures (1457°F (827°C)) for an extended period (≈2,700 hours). However, preflight verification of SD systems components, such as the PLR, require operation at ambient pressure and high temperatures. Therefore, the purpose of this research was to evaluate the durability of the alumina-titania coating at high temperature in air. Fifteen of sixteen alumina-titania coated Incoloy samples were exposed to high temperatures (600°F (316°C) to 1500°F (816°C)) for various durations (2 to 32 hours). Samples were characterized prior to, and after, heat treatment for reflectance, solar absorptance, room temperature emittance and emittance at 1200°F (649°C). Samples were also examined to detect physical defects and to determine surface chemistry using optical microscopy, scanning electron microscopy, operated with an energy dispersive spectroscopy (EDS) system, and x-ray photoelectron spectroscopy (XPS). Visual examination of the heat-treated samples showed a whitening of samples exposed to temperatures of 1000°F (538°C) and above. Correspondingly, the optical properties of these samples had degraded. A sample exposed to 1500°F (816°C) for 24 hours had whitened and the thermal emittance at 1200°F (649°C) had decreased from the non-heat treated value of 0.94 to 0.62. The coating on this sample had become embrittled, with spalling off the substrate noticeable at several locations. Based on this research it is recommended that preflight testing of SD components with alumina-titania coatings be restricted to temperatures no greater than 600°F (316°C) in air to avoid optical degradation. Moreover, components with the alumina-titania coating are likely to experience optical property degradation with direct atomic oxygen exposure in space.

INTRODUCTION

Solar dynamic space power systems require durable, high emittance surfaces on a number of critical components such as on the heat receiver interior surfaces and on the PLR elements. An alumina-titania coating, which has been evaluated for SD heat receiver canister applications, has been chosen for PLR (an electrical sink for excess power from the turboalternator/compressor (Shaltens, 1995)) applications due to its demonstrated high emittance and high temperature durability in vacuum (de Groh, 1994). Under high vacuum conditions ($\leq 10^{-6}$ torr) the alumina-titania coating was found to be durable maintaining a nearly constant emittance at a temperature of 1520°F (827°C) for ≈2,700 hours as shown in Figure 1 (de Groh, 1994). This coating has been successfully applied to the 500 thermal energy storage containment canisters inside the heat receiver, and the PLR radiator (Figure 2), of the 2 kW solar dynamic ground test demonstrator (Figure 3) at NASA LeRC. The SD GTD has successfully operated for over 500 hours in a LeRC's large thermal/vacuum space environment facility, demonstrating the feasibility of solar dynamic power generation for space applications (Shaltens, 1996a).

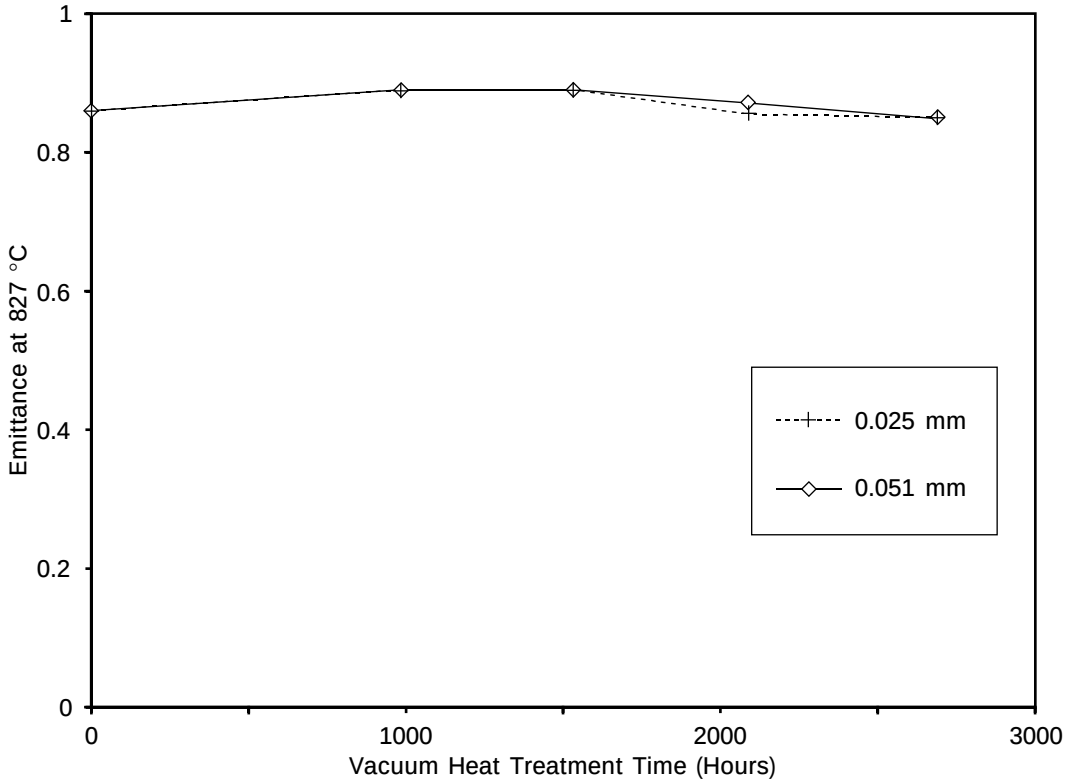

FIGURE 1. Emittance versus vacuum heat treatment (at 827 °C and 10^{-6} torr) time for two thicknesses of alumina-titania coating on Haynes 188 substrates.

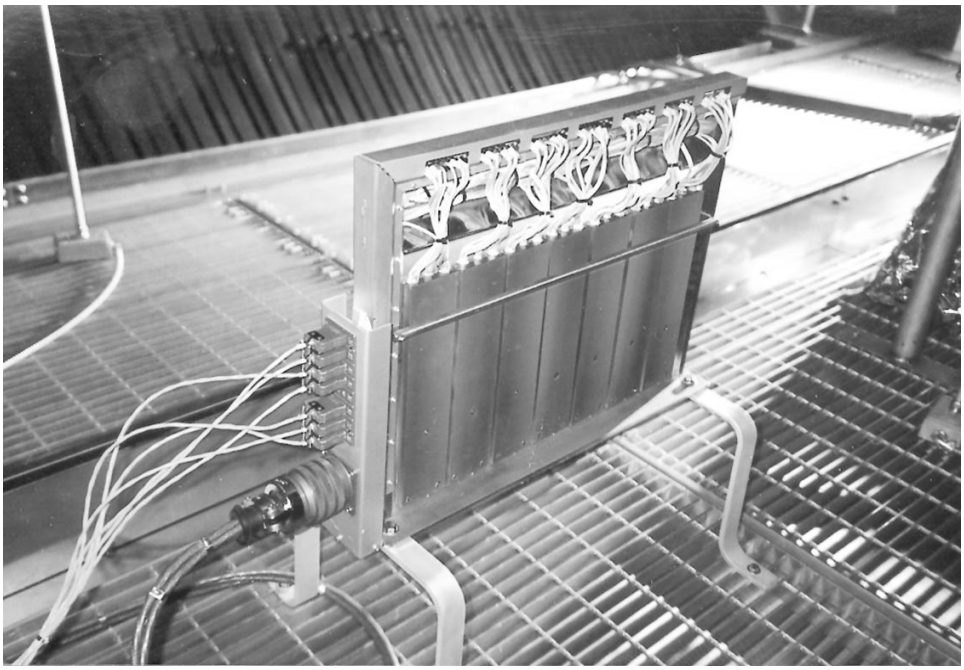

FIGURE 2. Parasitic load radiator of NASA Lewis' SD GTD system.

Although the alumina-titania high emittance coating has functioned successfully in the SD GTD system, SD flight PLR hardware verification will require additional ambient pressure operation. This is needed in order to verify that the electrical system and alternator control software function properly. PLR operation temperatures have been estimated to be around 1200°F (649°C) under full load, with a maximum design temperature of 1500°F (816°C). If operation of the PLR in air causes a decrease in emittance, the PLR will operate at a higher temperature than desired while on-orbit. This could shorten the PLR life, and possibly damage the system's elements. Therefore, the joint United States/Russian SD flight demonstration (SDFD) project (Wanhainen, 1995) decided to test the high temperature durability of the alumina-titania coating in air. If degradation of the high emittance surface occurs with high temperature air exposure, it is critical to determine what limitations (time/temperature) should be placed on operating the PLR in air prior to flight. It is also important to determine whether any damage could be reversed through procedures such as heat treatment in a reduction environment.

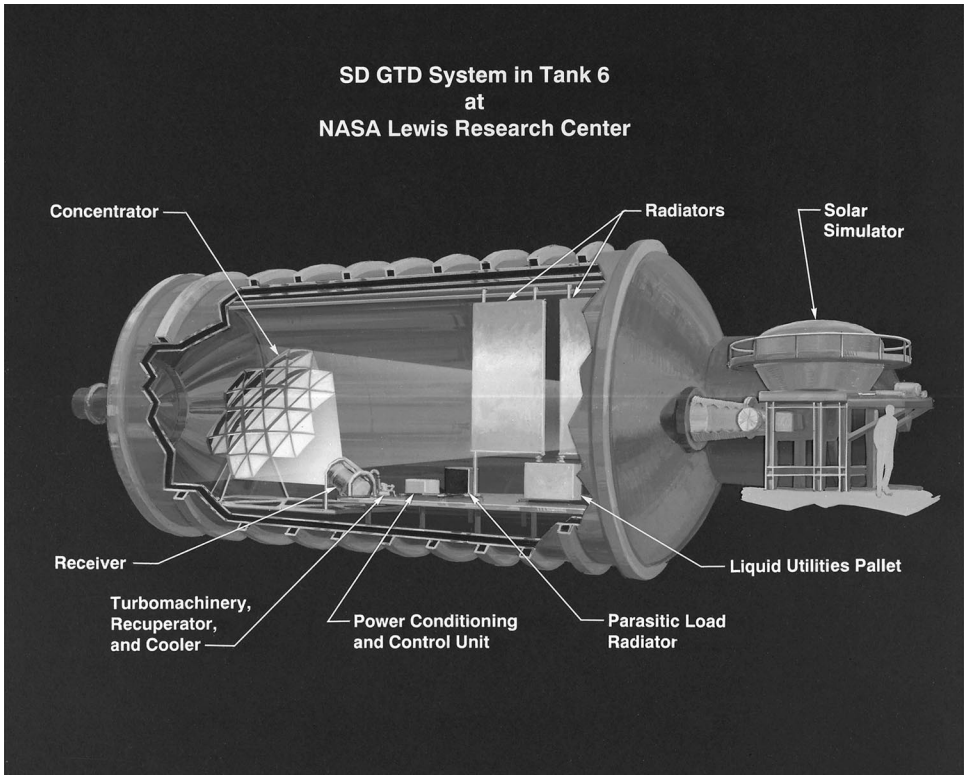

FIGURE 3. SD GTD system in the large space thermal/vacuum system at NASA Lewis Research Center.

MATERIALS AND EXPERIMENTAL PROCEDURES

AlliedSignal Aerospace, the contractor for the power conversion unit and heat receiver, prepared sixteen test coupons for durability evaluation. From an Incoloy 800 substrate, samples were cut to 1.2" x 1.2" squares and detonation gun flame-sprayed at Praxair with 0.001"-0.002" thick alumina-titania coatings. The alumina-titania composition consisted of 60 wt % Al_2O_3 - 40 wt % TiO_2. Fifteen samples were heat treated in air for various durations and temperatures. Twelve samples were exposed to the array of conditions corresponding to 600°F (316°C), 1000°F (538°C) and 1250°F (677°C) for 4, 8, 24 and 32 hours. Three samples were exposed to 1500°F (816°C) for 2, 8 and 24 hours. One sample was left untreated as a control. Photographs (1.2X) and optical micrographs (12X and 101X) were taken. The samples were characterized for their optical properties (reflectance, absorptance and emittance). In addition, scanning electron micrographs, EDS spectra and XPS spectra were obtained for both the control sample and the sample exposed to the highest temperature for the longest duration (PLR15) to compare surface morphologies and chemistries.

Optical Properties

Solar integrated total (ρ_t), diffuse (ρ_d), and specular reflectance (ρ_s) were obtained using a Perkin-Elmer λ-9 Spectrophotometer operated with a 150 mm integrating sphere. Reflectance was measured from 250 nm to 2500 nm and the data were convoluted into the air mass zero solar spectrum to obtain solar integrated values. With opaque samples like these, the solar absorptance (α_s) can be calculated by subtracting the integrated solar total reflectance from one.

Values for emittance at room temperature ($\varepsilon_{77°F}$) were obtained using a Gier Dunkel DB-100 infrared reflectometer which provides an integrated reflectance value which is then subtracted by 1 to get $\varepsilon_{77°F}$. Integrated thermal emittance at 1200°F (649°C) values were calculated using the spectral total reflectance obtained from the λ-9. Emittance was calculated by first extrapolating the last 250 data points of the total reflectance curve (2250 data points) to higher wavelengths to encompass 95% of the blackbody spectrum at 1200°F (649°C). This temperature was chosen because it was estimated to be the maximum operating temperature of the PLR on-orbit. Based on the shape of the curve for the last 250 data points from the λ-9, their root mean square (RMS) average was chosen for extrapolation. Next, the spectral absorptance was calculated by subtracting the spectral reflectance from 1. Using Kirchhoff's law ($\varepsilon_\lambda = \alpha_\lambda$) the spectral emittance was obtained (Siegel, 1981). The spectral emittance curve was then convoluted into the blackbody curve for 1200°F (649°C) to give integrated thermal emittance at 1200°F ($\varepsilon_{1200°F}$). While the extrapolation introduces uncertainty in the absolute values, the data still yields valuable information by showing the overall change in emittance that was introduced due to the heat treatment.

Surface Characterization

Overall photographs were taken with a Polaroid Landcamera. Optical micrographs were taken on an Olympus SZH Stereo-zoom microscope. Electron micrographs were taken on a JEOL 840 Scanning Electron Microscope (SEM). Energy dispersive x-ray spectrums were obtained using a Hitachi S-4700 Field Emission Electron Microscope operated at 20 kV with an EDAX DX Prime system. X-ray photoelectron spectroscopy spectra were obtained on a VG Scientific ESCA Lab MkII using nonmonochromatized Al K-alpha x-rays. The analyzed spot was 1 mm in diameter. The peak areas were converted to atomic-percent (AT %) using sensitivity factors supplied by the manufacturer. For surface cleaning or depth profiling, the sample was sputter-etched with 2.5 keV Ar ions rastered over a 3.5 mm by 3.5 mm area.

RESULTS AND DISCUSSION

The control and heat-treated samples are shown in Figure 4. Visual inspection of the samples indicates little degradation of the originally blue-black surfaces at 600°F (316°C). At higher temperatures, pronounced whitening of the surface coating is noticeable. At 1500°F (816°C), the coupons are essentially white and on sample PLR15, exposed for 24 hours at 1500°F, embrittlement and spalling of the coating is observable (see Figure 5).

Figure 6 shows the influence of heat treatment on the reflectance of the PLR samples. Figure 7 shows the influence of heat treatment on the emittance at 1200°F. The results indicate that treatment at 600°F (316°C) and 1000°F (538°C) did not induce a significant change in reflectance or emittance. Only after heat treatment for 8 hours at a temperature of 1250°F (677°C) did a significant change occur. Table 1 is a summary of the optical data obtained. This table shows that the emittance at 1200°F of sample PLR15 (heated for 24 hours at 1500°F) is approximately 32% lower than the emittance for the control sample. It is interesting to note that heat treatment does not affect room temperature emittance much, yet has a large effect on the high temperature emittance. This is reflected in the λ-9 spectra, where larger increases in reflectance are seen in the visible and less in the infrared wavelengths.

Electron microscopy revealed a significant change in the surface morphology of the coating with heating at 1500°F (816°C) for 24 hours, as can been seen in Figure 8. Between the two cases, the heat-treated sample appears to be flatter, with micro-cracks visible in the coating. The unheated coating remained electrically neutral during SEM imaging, but the heated sample became electrically charged, indicating a decrease in the electrical conductivity with heating.

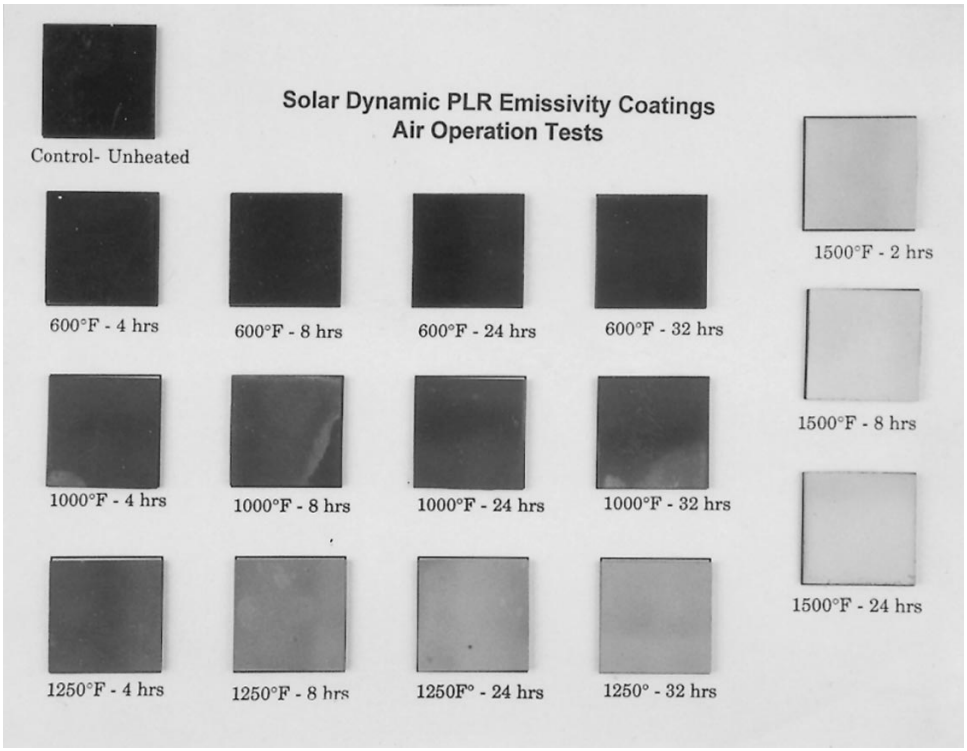

FIGURE 4. Alumina-titania coated Incoloy samples after heat treatment at various temperatures and times in air.

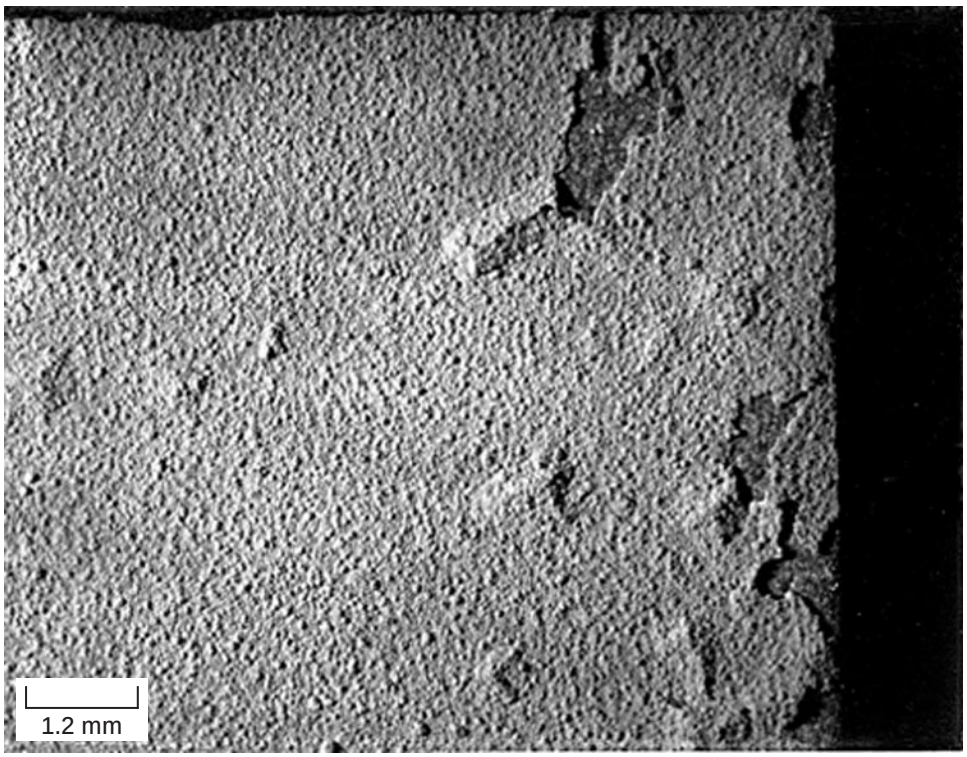

FIGURE 5. Sample PLR 15 showing coating embrittlement and spalling after heat treatment at 1500 °F (816 °C) for 24 hours.

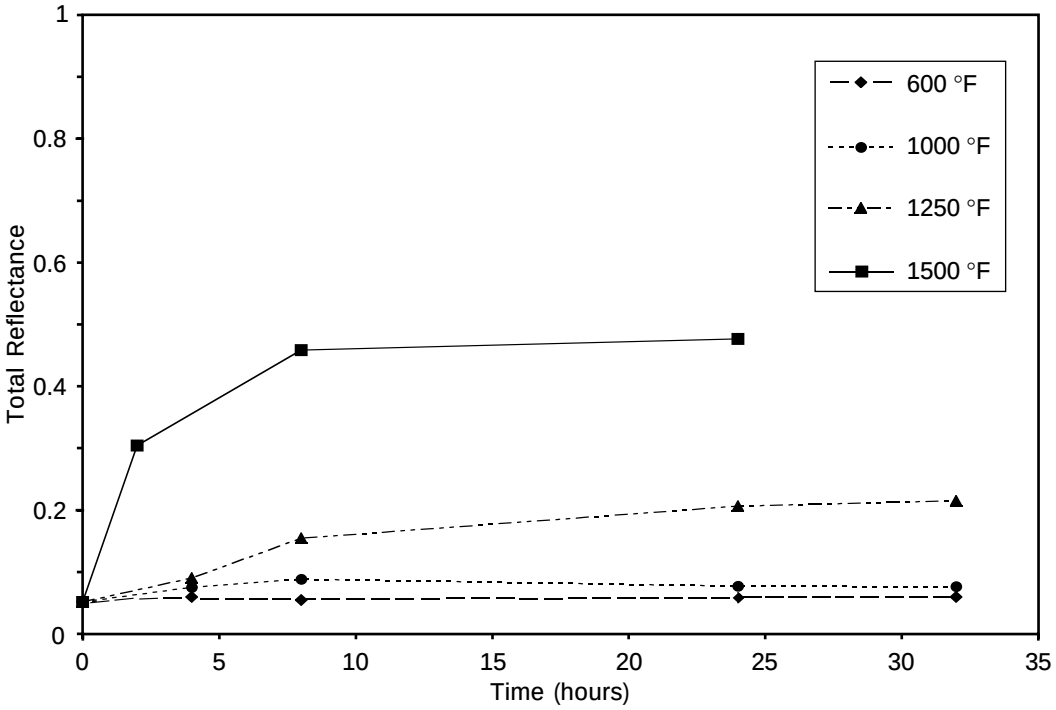

FIGURE 6. Total reflectance versus time for alumina-titania coated Incoloy samples exposed to heat treatment at various temperatures.

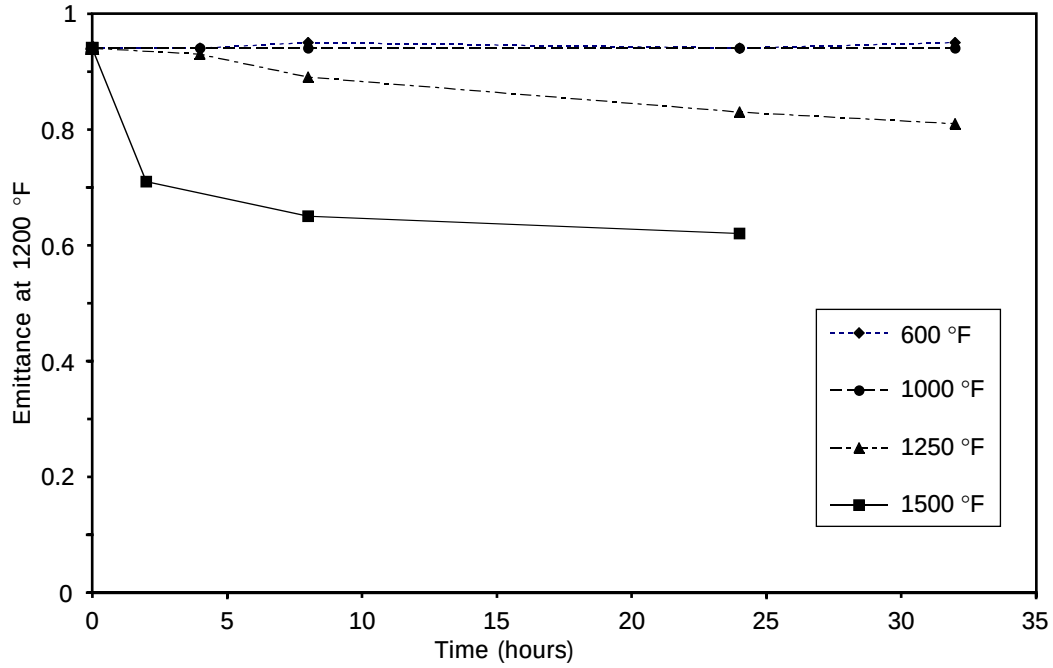

FIGURE 7. Emittance at 1200 °F (649 °C) versus time for alumina-titania coated Incoloy samples exposed to heat treatment at various temperatures.

TABLE 1. Optical Properties of Heat Treated Samples

Sample	Treatment	ρ_t	α_s	$\varepsilon_{77\,°F}$	$\varepsilon_{1200\,°F}$
Control	UNTREATED	0.051	0.949	0.884	0.94
PLR1	4 hrs at 600 °F	0.059	0.941	0.889	0.94
PLR2	8 hrs at 600 °F	0.054	0.946	0.88	0.95
PLR3	24 hrs at 600 °F	0.058	0.942	0.875	0.94
PLR4	32 hrs at 600 °F	0.059	0.941	0.883	0.95
PLR5	4 hrs at 1000 °F	0.075	0.925	0.877	0.94
PLR6	8 hrs at 1000 °F	0.088	0.912	0.874	0.94
PLR7	24 hrs at 1000 °F	0.077	0.923	0.852	0.94
PLR8	32 hrs at 1000 °F	0.076	0.924	0.86	0.94
PLR9	4 hrs at 1250 °F	0.09	0.91	0.872	0.93
PLR10	8 hrs at 1250 °F	0.154	0.846	0.884	0.89
PLR11	24 hrs at 1250 °F	0.206	0.794	0.854	0.83
PLR12	32 hrs at 1250 °F	0.215	0.785	0.863	0.81
PLR13	2 hrs at 1500 °F	0.304	0.696	0.838	0.71
PLR14	8 hrs at 1500 °F	0.458	0.542	0.83	0.65
PLR15	24 hrs at 1500 °F	0.476	0.524	0.84	0.62

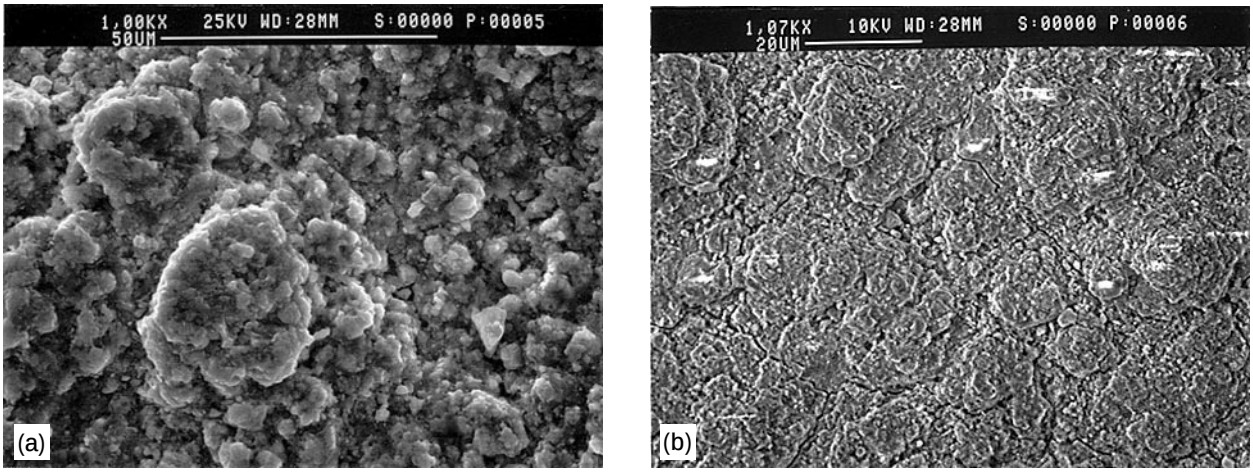

FIGURE 8. Surfaces morphologies of as-received and heat treated alumina-titania coated Incoloy samples. (a) Control sample (unheated). (b) Sample PLR15 heated at 1500 °F (816 °C) for 24 hours.

Because the heated samples appeared flatter in the SEM, the control sample and PLR15 were scanned for average roughness with a Dektak diamond stylus profilometer. The control sample had an average roughness of 5.6 ± 1.2 μm (3 scans), whereas the heated sample had an average roughness of 4.3 ± 0.9 μm (4 scans).

Initial XPS analyses indicated that the as-deposited coatings were very high in C (48.5 AT % at the surface), even at sputter depths that would normally remove surface C (20.1 AT % at 43 nm depth). Because the coating surface is very rough, the samples were decided to be sputter etched from 4 different angles for depth profiling, in an attempt to remove surfaces that otherwise might be shielded if ion beam etching occurred from a single direction. Surface surveys and depth profiling surveys (after etching from 4 directions) of the as-received coating and a heat-treated sample are listed in Table 2 and 3, respectively.

TABLE 2. XPS depth profile results for the as-deposited coating

Element	As-Deposited Coating				
	Peak Center (eV)	Surface AT %	1 nm depth AT%	43 nm depth AT %	147 nm depth AT%
C	285.85	53.730	60.634	27.470	12.116
O	530.91	35.762	25.670	45.565	54.161
Al	74.00	5.455	6.927	15.566	20.994
Ti	458.72	3.111	3.435	7.758	9.882
Pb	138.58	0.575	0.492	0.505	0.285
Zn	1021.60	0.476	0.247	0.092	0.066
Fe	712.00	0.204	1.106	0.914	1.259
Zr	182.5	0.192	0.516	0.642	0.494
Ca	347.14	0.160	0.349	0.675	0.474
Cu	932.60	0.160	0.266	0.191	0.229
K	294.85	0.144	0.208	0.0	0.0
Ag	368.00	0.033	0.083	0.0	0.039
W	247.56	0.0	0.065	0.622	0.0

TABLE 3. XPS depth profile results for heat treated sample PLR-15

Element	Heat Treated Sample (PLR-15)				
	Peak Center (eV)	Surface AT %	1 nm depth AT%	43 nm depth AT %	147 nm depth AT%
C	284.85	14.988	7.540	1.260	0.688
O	530.91	56.863	60.347	62.889	62.324
Al	74.00	15.188	17.041	19.986	21.828
Ti	458.72	6.328	7.855	11.247	11.427
Pb	138.58	0.233	0.472	0.324	0.098
Zn	1021.60	0.027	0.135	0.084	0.0
Fe	712.00	0.720	0.611	0.394	0.790
Zr	182.5	0.724	0.659	0.843	0.768
Ca	347.14	0.220	0.252	0.332	0.390
Cu	932.60	0.110	0.014	0.004	0.0
K	292.65	0.806	0.915	0.335	0.216
Ag	368.00	0.666	0.674	0.271	0.139
W	247.56	3.127	3.487	2.031	1.331

Even after ion beam etching from 4 directions, the as-deposited coating was found to be high in C (60.3 AT % at 1 nm depth). After etching to a depth of 147 nm, there was still a substantial amount of C present in the coating (12.1 AT %). It is possible that carbon was incorporated in the coating from the acetylene gas used during the deposition process. Small amounts of other "trace" elements such as Pb, Zn, Fe, Zr, Ca, Cu, K and Ag were detected. These trace elements are also likely due to the detonation gun spray process. The Al and Ti peak centers (74.00 and 458.72, respectively) are consistent with metal oxides and not elemental metals (Moulder). At a depth of 147 nm there was 54 AT % O, 21.0 AT % Al and 9.9 AT % Ti. It should be noted that after sputter etching to a depth of 147 nm where the C was mostly removed, the surface still appeared black.

Heating the coating in air at 1500°F for 24 hours resulted in a significant loss of C (from 53.7 to 15.0 AT % at the surface) and a gain in O (from 35.8 to 56.8 AT %). The original "trace" elements changed in AT % slightly with heat treating, and a significant amount of W is present in the heat-treated sample (3.1 AT %). The binding-energy peak centers for Al and Ti did not change. But, a change of the Ti peak shape (two distinct peaks, one at 455.6 eV) was observed for the heat-treated spectra at 147 nm depth, as compared to the as-received coating. By curve fitting for TiO_2 and TiO, the as-received coating appears to have a TiO_2/TiO ratio of ≈1, and the heat-treated sample has a TiO_2/TiO ratio of ≈1.6. It should be noted that TiC also has a peak at ≈455.0 eV (Moulder). It has been proposed that a change in oxidation state is responsible for the optical property changes. Color changes associated with changes in the stoichiometry of alumina have been reported by Arghiropoulos. The loss of oxygen during the coating process is believed to cause the white oxides to turn

black. At a depth of 147 nm the heat-treated sample has only 0.7 AT % C compared to 62.3 AT % O, 21.8 AT % Al and 11.4 AT % Ti. These quantities are closer the those expected for Al_2O_3 (60%) – TiO_2 (40%) which are 61.9 AT % O, 28.6 AT % Al and 9.5 AT % Ti.

Energy dispersive spectroscopy, which is a deeper penetrating analytical technique (1-5 μm) than XPS (10-100 Å), was conducted on the XPS sputter etched control sample to check if C was present in the bulk of the coating. The as-received coating had 10.2 AT % C, 54.5 AT % O, 24.9 AT % Al and 10.4 AT % Ti. This is in good agreement with the XPS analysis of the as-deposited coating at a depth of 147 nm. A SiC fiber was analyzed at the same time to verify accuracy of the system. Carbon, which is present in the as-received coating has been oxidized and is thus not present in the bulk of the heat-treated sample, and therefore may also play a minor role in the optical properties of the coating.

The emittance data suggests that ambient high temperature exposure is not a problem unless the coating is heated to temperatures above 1000°F (538°C). Although no significant optical property changes were observed until 8 hours of exposure at 1250°F (677°C), visible whitening and small changes in the reflectance and hence absorptance were observed for samples exposed to 1000°F (538°C). This implies that the coating would not be stable at this temperature for long durations. Also, at around 1000°F (538°C) titania undergoes a phase change which might exacerbate oxidation and cause spalling of the coating (Leissler, private communication). This research indicates that the high temperature optical property degradation of the alumina-titania coating is not likely reversible. The loss of C (which may play a minor role in optical properties) from the coating is a permanent loss. It was proposed that if the oxides do gain some oxygen during heating, exposure to a reduction environment apparently could reverse the degradation caused by oxidation. Yet, Praxair, the coating supplier, has attempted reversal of the coating degradation using a hydrogen reduction environment but was unsuccessful (Naim, private communication). Even if it was successful, this would not reverse the embrittlement and spalling which occurred at 1500°F (816°C) after 24 hours. Arghiropoulos found reversible stoichiometric transformations in alumina at 932°F (500°C), and irreversible transformations at 1832°F (1000°C).

Based on this research it is recommended that components coated with the alumina-titania coating be operated at temperatures of 600°F (316°C) or below in air. It is possible that there are temperatures above 600°F and below 1000°F for which the coating would be stable. However, further tests are necessary to determine a more precise failing temperature and temperatures in this range were not investigated in this test program. The SD ground test demonstrator PLR which is coated with the alumina-titania coating, has been operated for over 365 hours in air (Shaltens, 1996b). Even at the maximum load (2kW) the heat generated by the PLR appears to be moderate (personnel could put their hands near it during operation (Shaltens, private communication), and there has been no observable degradation of the coating. This indicates that PLR operation temperatures might be lower than estimated and thus would not pose a threat to the optical properties of the coating if operated in air.

Finally, it should be noted that based on this research, atomic oxygen (which is more reactive than molecular oxygen) degradation is likely to be a concern for operation on orbit because the PLR panels will be directly exposed to the low Earth orbit environment, unlike the heat receiver interior. The atomic oxygen durability of the coating therefore needs to be assessed prior to use of this coating on surfaces exposed to atomic oxygen impingement. Preliminary data obtained on alumina-titania coated Haynes 188 substrates provided evidence of a significant decrease in optical properties with high temperature (1520°F (827°C)) atomic oxygen exposure, as shown in Figure 9. The total plasma asher exposure (160 hours) was equal to a Kapton effective fluence of 5.8×10^{20} atoms/cm^2 (or a performance requirement of 0.2 years of sweeping ram atomic oxygen exposure on the International Space Station). Testing has not been conducted to see if lower temperature atomic oxygen exposure would also cause optical property degradation.

SUMMARY AND CONCLUSIONS

Solar dynamic power system components, such as the parasitic load radiator, which are covered with a high temperature, vacuum durable, high emittance alumina-titania coating need ambient pressure operation in order to verify proper system electrical function and to check out alternator control software of actual flight hardware. Sixteen samples of alumina-titania coated Incoloy were prepared for durability evaluation at high temperature in air. Fifteen samples were heat treated at various temperatures for different durations. Visual examination of the samples indicated a whitening of the originally blue-black coating upon exposure to 1000°F and higher. Degradation of the optical properties of the samples exposed

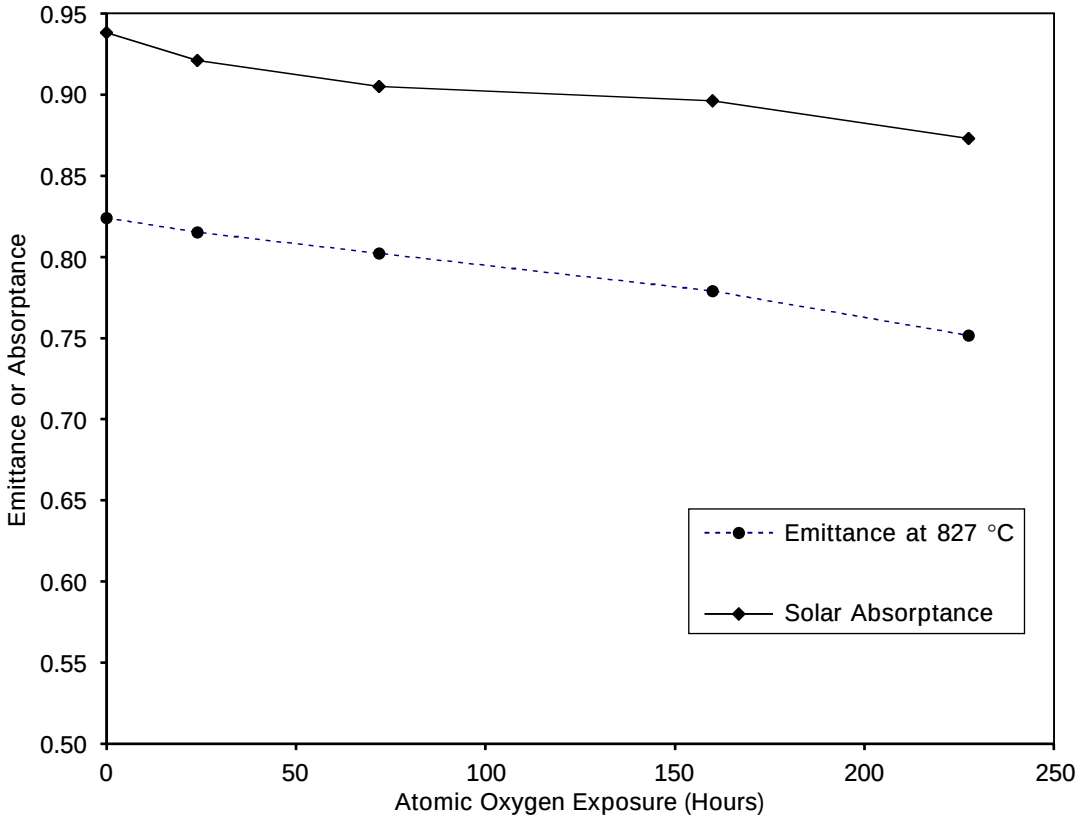

FIGURE 9. Optical property degradation of atomic oxygen exposed alumina-titania coated Haynes 188 samples at elevated temperature.

to 1000°F or higher was verified through reflectance, absorptance and emittance changes. A sample exposed to 1500°F for 24 hours appeared white, the thermal emittance at 1200°F decreased from 0.94 to 0.62, and the coating became embrittled with spalling from the substrate observable in several locations. Based on this research it is recommended that components with the alumina-titania coating be operated at temperatures of 600°F (316°C) or below in air prior to space flight. Finally, the atomic oxygen durability of the coating appears to be a significant concern and needs to be addressed prior to use of this coating on components directly exposed to the space environment.

REFERENCES

Arghiropoulos, B., et al., "Reactivity of Non-Stoichiometric Black Alumina," Proceedings of the 4th International Symposium on the Reactivity of Solids, Amsterdam, May 30-June 4, 1960, p. 524-539.

de Groh, K. K., Roig, D. M., Burke, C. A. and Shah, D. R., "Performance and Durability of High Emittance Heat Receiver Surfaces for Solar Dynamic Power Systems, NASA TM–106549, 1994.

Leissler, G., (NYMA, Inc.) Private Communication.

Moulder, J. F., et. al., *Handbook of X-Ray Photoelectron Spectroscopy*, Perkin-Elmer Corp., USA.

Naim, M. (Praxair), Private Communication.

Shaltens, R. K. & Boyle, R. V., "Initial Results from the Solar Dynamic (SD) Ground Test Demonstration (GTD) Project at NASA Lewis," 30th IECEC, Orlando Florida, July 31-August 4, 1995, IECEC-95-421, also NASA TM–107004.

Shaltens, R. K. and Mason, L. S., "500 Hours of Operational Experience from a Solar Dynamic Power System," 31st Intersociety Energy Conversion Engineering Conference, Washington, D.C., August 11-16, 1996a, IECEC-96568; also NASA TM–107294.

Shaltens, R. K., and L. S. Mason, "Early Results From Solar Dynamic Space Power System Testing," NASA TM–107252, 1996b.

Shaltens, R. K., (NASA Lewis Research Center) Private Communication.

Siegel, R. and Howell J. R., *Thermal Radiation Heat Transfer*, Hemisphere Publishing Corporation, Washington, 2nd ed., 1981.

Wanhainen, J. S. and Tyburski, T. E., "Joint US/Russian Solar Dynamic Flight Demonstration Project Plan," IECEC Paper No. AP-176, ASME, 1995, pp. 417-420

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13. ABSTRACT ***(Maximum 200 words)***

Solar dynamic (SD) space power systems require durable, high emittance surfaces on a number of critical components, such as heat receiver interior surfaces and parasitic load radiator (PLR) elements. To enhance surface characteristics, an alumina-titania coating has been applied to 500 heat receiver thermal energy containment canisters and the PLR of NASA Lewis Research Center's (LeRC) 2 kW SD ground test demonstrator (GTD). The alumina-titania coating was chosen because it had been found to maintain its high emittance under vacuum ($\leq 10^{-6}$ torr) at high temperatures (1457°F (827°C)) for an extended period (≈2,700 hours). However, preflight verification of SD systems components, such as the PLR, require operation at ambient pressure and high temperatures. Therefore, the purpose of this research was to evaluate the durability of the alumina-titania coating at high temperature in air. Fifteen of sixteen alumina-titania coated Incoloy samples were exposed to high temperatures (600°F (316°C) to 1500°F (816°C)) for various durations (2 to 32 hours). Samples were characterized prior to, and after, heat treatment for reflectance, solar absorptance, room temperature emittance and emittance at 1200°F (649°C). Samples were also examined to detect physical defects and to determine surface chemistry using optical microscopy, scanning electron microscopy, operated with an energy dispersive spectroscopy (EDS) system, and x-ray photoelectron spectroscopy (XPS). Visual examination of the heat-treated samples showed a whitening of samples exposed to temperatures of 1000°F (538°C) and above. Correspondingly, the optical properties of these samples had degraded. A sample exposed to 1500°F (816°C) for 24 hours had whitened and the thermal emittance at 1200°F (649°C) had decreased from the non-heat treated value of 0.94 to 0.62. The coating on this sample had become embrittled, with spalling off the substrate noticeable at several locations. Based on this research it is recommended that preflight testing of SD components with alumina-titania coatings be restricted to temperatures no greater than 600°F (316°C) in air to avoid optical degradation. Moreover, components with the alumina-titania coating are likely to experience optical property degradation with direct atomic oxygen exposure in space.

14. SUBJECT TERMS

Solar dynamic system; Thermal emittance; Solar absorptance; Alumina-titania; Heat treatment; Ambient pressure; High temperature durability; Solar dynamic ground test demonstrator

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